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मानक

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Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

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Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 3400-4 (2012): Methods of Test for Vulcanized Rubbers,
Part 4: Accelerated Ageing and Heat Resistance [PCD 13:
Rubber and Rubber Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
वल्कनीकृत रबड़ के लिए परीक्षण पद्धतियाँ
भाग 4 त्वरित काल प्रभावन और ताप प्रतिरोधन
(तीसरा पुनरीक्षण)

Indian Standard
METHODS OF TEST FOR VULCANIZED RUBBER
PART 4 ACCELERATED AGEING AND HEAT RESISTANCE
(*Third Revision*)

ICS 83.060

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Rubber and Rubber Products Sectional Committee, PCD 13

NATIONAL FOREWORD

This Indian Standard (Part 4) (Third Revision) which is identical with ISO 188 : 2011 'Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Rubber and Rubber Products Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1965 and subsequently revised in 1977 and 1987. This revision has been undertaken to consider the developments that have taken place since the last revision, the committee has now decided to adopt this standard under dual numbering system. This third revision is being carried out to align it with the latest published International Standard on the subject.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respective places are listed below along with their degree of equivalence for the editions indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 37 : 2011 Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties	IS 3400 (Part 1) : 2012 Methods of test for vulcanized rubber: Part 1 Tensile stress-strain properties (<i>third revision</i>)	Identical
ISO 48 : 1994 ¹⁾ Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)	IS 3400 (Part 2) : 2003 Methods of test for vulcanized rubber: Part 2 Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD) (<i>third revision</i>)	do

The technical committee has reviewed the provisions of the following International Standards referred in this adopted standard and has decided that they are acceptable for use in conjunction with this standard:

<i>International Standard</i>	<i>Title</i>
ISO 18899 : 2004	Rubber — Guide to the calibration of test equipment
ISO 23529 : 2004	Rubber — General procedures for preparing and conditioning test pieces for physical test methods

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

¹⁾Since revised in 2007.

Introduction

Accelerated ageing and heat resistance tests are designed to estimate the relative resistance of rubber to deterioration with the passage of time. For this purpose, the rubber is subjected to controlled deteriorating influences for definite periods, after which appropriate properties are measured and compared with the corresponding properties of the unaged rubber.

In accelerated ageing, the rubber is subjected to a test environment intended to produce the effect of natural ageing in a shorter time.

In the case of heat resistance tests, the rubber is subjected to prolonged periods at the same temperature as that which it will experience in service.

Two types of method are given in this International Standard, namely an air-oven method using a low air speed and an air-oven method using forced air ventilation for a high air speed.

The selection of the time, temperature and atmosphere to which the test pieces are exposed and the type of oven to use will depend on the purpose of the test and the type of polymer.

In air-oven methods, deterioration is accelerated by raising the temperature. The degree of acceleration thus produced varies from one rubber to another and from one property to another.

Degradation can also be accelerated by air speed. Consequently, ageing with different ovens can give different results.

Consequences of these effects are:

- a) Accelerated ageing does not truly reproduce under all circumstances the changes produced by natural ageing.
- b) Accelerated ageing sometimes fails to indicate accurately the relative natural or service life of different rubbers; thus, ageing at temperatures greatly above ambient or service temperatures may tend to equalize the apparent lives of rubbers, which deteriorate at different rates in storage or service. Ageing at one or more intermediate temperatures is useful in assessing the reliability of accelerated ageing at high temperatures.
- c) Accelerated ageing tests involving different properties may not give agreement in assessing the relative lives of different rubbers and may even arrange them in different orders of merit. Therefore, deterioration should be measured by the changes in property or properties which are of practical importance, provided that they can be measured reasonably accurately.

Air-oven ageing should not be used to simulate natural ageing which occurs in the presence of either light or ozone when the rubbers are stretched.

To estimate lifetime or maximum temperature of use, tests can be performed at several temperatures and the results can be evaluated by using an Arrhenius plot or the Williams Landel Ferry (WLF) equation as described in ISO 11346^[2].

Indian Standard
METHODS OF TEST FOR VULCANIZED RUBBER
PART 4 ACCELERATED AGEING AND HEAT RESISTANCE
(Third Revision)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies accelerated ageing or heat resistance tests on vulcanized or thermoplastic rubbers. Two methods are given:

Method A: air-oven method using a cell-type oven or cabinet oven with low air speed and a ventilation of 3 to 10 changes per hour;

Method B: air-oven method using a cabinet oven with forced air circulation by means of a fan and a ventilation of 3 to 10 changes per hour.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 37, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

ISO 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 18899:2004, *Rubber — Guide to the calibration of test equipment*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Principle

3.1 General

Test pieces are subjected to controlled deterioration by air at an elevated temperature and at atmospheric pressure, after which specified properties are measured and compared with those of unaged test pieces.

The physical properties concerned in the service application should be used to determine the degree of deterioration but, in the absence of any indication of these properties, it is recommended that tensile strength, stress at intermediate elongation, elongation at break (in accordance with ISO 37) and hardness (in accordance with ISO 48) be measured.

3.2 Accelerated ageing by heating in air

In this method, the test pieces are subjected to a higher temperature than the rubber would experience in service in order to produce the effects of natural ageing in a shorter time.

3.3 Heat resistance test

In this method, the test pieces are subjected to the same temperature as they would experience in service.

4 Apparatus

4.1 Air oven

4.1.1 General

The oven shall be of such a size that the total volume of the test pieces does not exceed 10 % of the free space in the oven. Provision shall be made for suspending test pieces so that they are at least 10 mm from each other and, in cabinet ovens and ovens with forced air circulation, at least 50 mm from the sides of the oven.

The temperature of the oven shall be controlled so that the temperature of the test pieces is kept within the specified tolerance for the specified ageing temperature (see Clause 8) for the whole ageing period. A temperature sensor shall be placed inside the heating chamber close to the samples to record the actual ageing temperature.

No copper or copper alloys shall be used in the construction of the heating chamber.

Provision shall be made for a slow flow of air through the oven of not less than three and not more than ten air changes per hour.

Care shall be taken to ensure that the incoming air is heated to within ± 1 °C of the temperature of the oven before coming in contact with the test pieces.

The ventilation (or air change rate) can be determined by measuring the volume of the oven chamber and the flow of air through the chamber.

NOTE To ensure good precision when doing ageing and heat resistance tests, it is very important to keep the temperature uniform and stable during the test and to verify that the oven used is within the temperature limits with regard to time and space. Increasing the air speed in the oven improves temperature homogeneity. However, air circulation in the oven and ventilation influence the ageing results. With a low air speed, accumulation of degradation products and evaporated ingredients, as well as oxygen depletion, can take place. A high air speed increases the rate of deterioration, due to increased oxidation and volatilization of plasticizers and antioxidants.

4.1.2 Cell-type oven

The oven shall consist of one or more vertical cylindrical cells having a minimum height of 300 mm. The cells shall be surrounded by a thermostatically controlled good heat transfer medium (aluminium block, liquid bath or saturated vapour). Air passing through one cell shall not enter other cells.

Provision shall be made for a slow flow of air through the cell. The air speed shall depend on the air change rate only.

4.1.3 Cabinet oven

This shall comprise a single chamber without separating walls. Provision shall be made for a slow flow of air through the oven. The air speed shall depend on the air change rate only, and no fans are allowed inside the heating chamber.

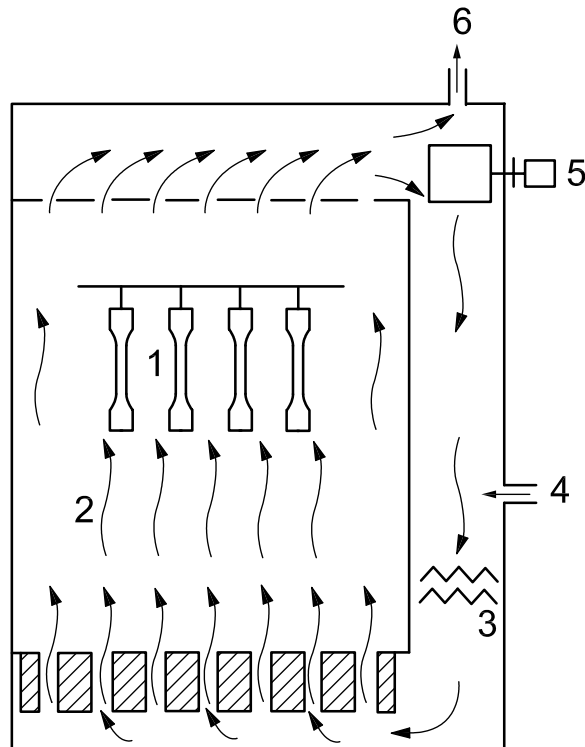
4.1.4 Oven with forced air circulation

Either of the following two types shall be used:

- a) Type 1 oven with laminar air flow (see Figure 1).

The air flow through the heating chamber shall be as uniform and laminar as possible. The test pieces shall be placed with the smallest surface facing towards the air flow to avoid disturbing the air flow. The air speed shall be between 0,5 m/s and 1,5 m/s.

The air speed near the test pieces can be measured by means of an anemometer.



Key

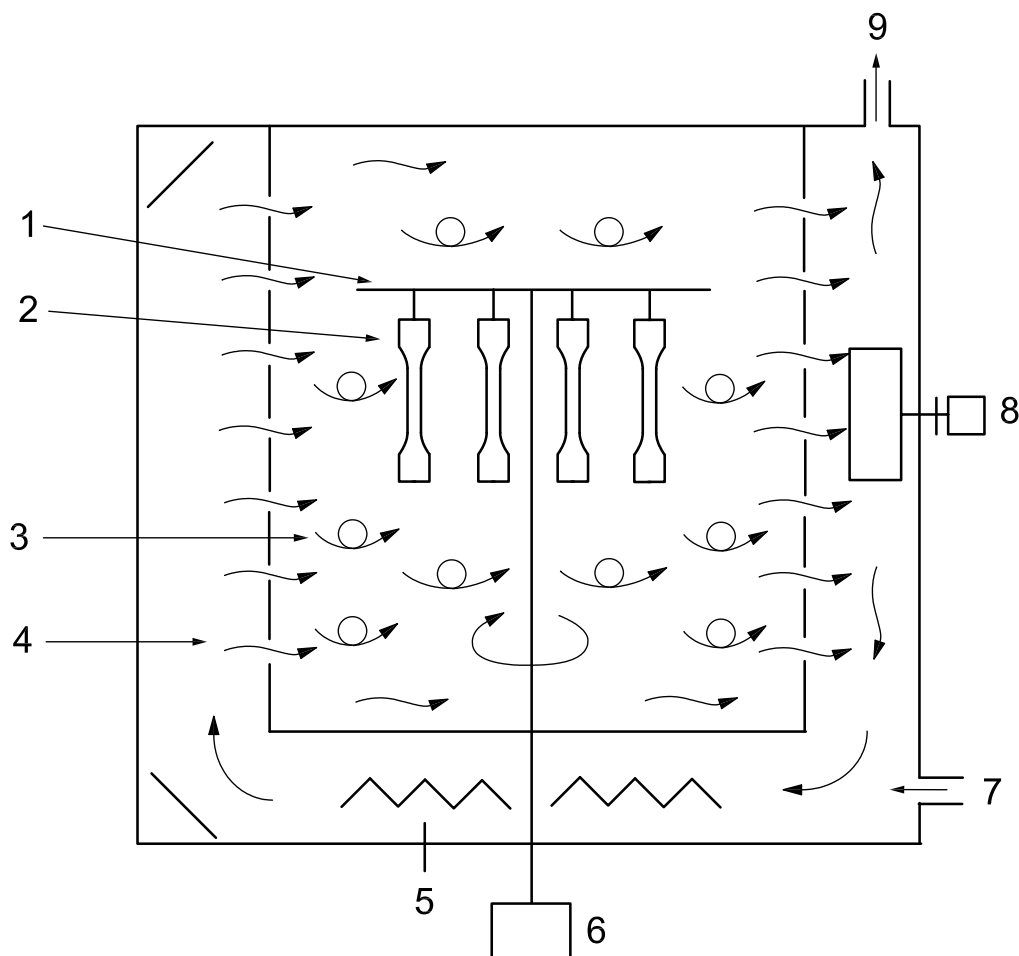
- 1 test pieces
- 2 laminar air flow
- 3 heating element
- 4 air inlet
- 5 air blower
- 6 air outlet

Figure 1 — Type 1 oven with laminar air flow

- b) Type 2 oven with turbulent air flow (see Figure 2).

The air entering from a side-wall air-inlet into the heating chamber is turbulent around the test pieces, which are suspended on a carrier rotating at a speed of five to ten rotations per minute so that they are exposed to the heating air as uniformly as possible. The average air speed shall be $0,5 \text{ m/s} \pm 0,25 \text{ m/s}$.

The average air speed near the test pieces can be calculated from measurements made with an anemometer at nine different positions (see Figure A.1 in Annex A). A suitable method of measurement is described in Annex A.



Key

- 1 test piece carrier
- 2 test pieces
- 3 turbulent air flow
- 4 laminar air flow (inlet, outlet and near to wall)
- 5 heating element
- 6 motor
- 7 air inlet
- 8 air blower
- 9 air outlet

Figure 2 — Type 2 oven with turbulent air flow

5 Calibration

The test apparatus shall be calibrated in accordance with Annex D.

6 Test pieces

It is recommended that the accelerated ageing or heat resistance test be carried out on test pieces prepared and conditioned as required for the appropriate property tests, and not on complete products or sample sheets, and that their form be such that no mechanical, chemical or heat treatment will be required after ageing.

Only test pieces of similar dimensions and having approximately the same exposed areas shall be compared with each other. The number of test pieces shall be in accordance with the International Standard for the

appropriate property tests. The test pieces shall be measured before heating but, whenever possible, marking shall be carried out after heating as some marking inks can affect the ageing of the rubber.

Care shall be taken to ensure that the markings used to identify the test pieces are not applied in any critical area of the test piece and will not damage the rubber or disappear during heating.

Avoid simultaneous heating of different types of compound in the same oven, to prevent the migration of sulphur, antioxidants, peroxides or plasticizers. For this purpose, the use of individual cells is highly recommended. In order, however, to give some guidance for cases where it is not practicable to provide equipment with individual cells, it is recommended that only the following types of material be heated together:

- a) polymers of the same general type;
- b) vulcanizates containing the same type of accelerator and approximately the same ratio of sulphur to accelerator;
- c) rubbers containing the same type of antioxidant;
- d) rubbers containing the same type and amount of plasticizer.

7 Time interval between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements shall be observed.

For all normal test purposes, the minimum time between vulcanization and testing shall be 16 h. In cases of arbitration, the minimum time shall be 72 h.

For non-product tests, the maximum time between vulcanization and testing shall be four weeks and, for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time-interval.

For product tests, whenever possible, the time between vulcanization and testing shall not exceed three months. In other cases, tests shall be made within two months of the date of receipt by the purchaser of the product.

8 Ageing conditions (duration and temperature)

8.1 General

The period required to obtain a given degree of deterioration of the test pieces will depend upon the type of rubber under examination.

The ageing period used should preferably be such that deterioration of the test pieces will not be so great as to prevent determination of the final values of physical properties.

The use of high ageing temperatures may result in different degradation mechanisms than those which occur at service temperatures, thus invalidating the results.

It is crucial for the best results that the temperature be kept as stable as possible. Temperature tolerances in ISO 23529 are ± 1 °C up to and including 100 °C and ± 2 °C for 125 °C up to and including 300 °C. However, studies have shown that a 1 °C change in temperature corresponds to a 10 % difference in ageing time at an Arrhenius factor of 2, or 15 % at a factor of 2.5. This means that two laboratories carrying out ageing at 125 °C can have ageing times which differ by 60 % from each other and still be within the specification. To get accurate results, keep the temperature as accurate as possible by placing a calibrated temperature sensor close to the test pieces and use this to set the oven so that the temperature at this position is correct. Use the correction factor from the calibration certificate to get as close as possible to the true temperature.

8.2 Accelerated ageing

The duration of ageing and the ageing temperature shall be chosen in accordance with ISO 23529, as stated in the product specification or as agreed between the interested parties. The ageing shall be performed at atmospheric pressure.

8.3 Heat resistance test

The test duration and the temperature of test shall be chosen in accordance with ISO 23529, as stated in the product specification or as agreed between the interested parties. The temperature shall be representative of the service temperature and the heating shall be carried out at atmospheric pressure.

9 Procedure

Heat the oven to the operating temperature and place the test pieces in it. When using a cell-type oven, only one rubber or compound shall be placed in each cell. The test pieces shall be free from strain, freely exposed to air on all sides and not exposed to light.

When the heating period is complete, remove the test pieces from the oven and condition them for not less than 16 h and not more than 6 days in a strain-free condition in the atmosphere given in the appropriate test method for the particular property being studied.

10 Expression of results

The results shall be expressed in accordance with the International Standard for the appropriate property tests.

The test results for both the unaged and the aged test pieces shall be reported together, as well as, when appropriate, the percentage change in the value of the property measured as calculated from the formula:

$$\frac{x_a - x_0}{x_0} \times 100$$

where

x_0 is the value of the property before ageing;

x_a is the value of the property after ageing.

Express changes in hardness as the difference $x_a - x_0$.

11 Precision

See Annex B.

12 Test report

The test report shall include the following information:

a) sample details:

- 1) a full description of the sample and its origin,
- 2) details of the compound and its condition of cure, if known,
- 3) the time interval between forming and testing,
- 4) the method used to prepare the test pieces (e.g. moulding, cutting from the sample) and the location of the test pieces in the sample;

b) test method:

- 1) a reference to this International Standard,
- 2) the method used (A or B),
- 3) the properties determined and the type of test piece used;

c) test details:

- 1) the type of oven used,
- 2) the number of test pieces used,
- 3) whether accelerated ageing or a heat resistance test was carried out,
- 4) the temperature and duration of ageing,
- 5) details of any procedures not specified in this International Standard;

d) test results:

- 1) the individual values before and after ageing, expressed in accordance with the International Standards for the appropriate property tests,
- 2) the changes in the property values, expressed as a percentage or, for hardness, as the difference between the values;

e) the date of the test.

Annex A (informative)

Determination of the air speed in ovens with forced air circulation

A.1 Scope

This annex describes a method for determining the air speed in both type 1 and type 2 ovens.

A.2 Apparatus

A portable anemometer can be used.

A.3 Procedure

A.3.1 Air speed should be measured at nine positions at the level of the centre of a suspended test piece. For this purpose, prepare an at least 2 mm thick transparent plastic plate made of PVC [poly(vinyl chloride)] or PMMA [poly(methyl methacrylate)], of the same size as the door of the oven chamber, and drill three apertures, each big enough to allow an anemometer to be inserted in it, two located 70 mm from the left and right edge, respectively, and one at the mid-point between the two (see Figure A.1).

A.3.2 The measurement of the air speed should be carried out at a standard laboratory temperature.

A.3.3 Open the door of the chamber and fix the plastic plate in the door opening.

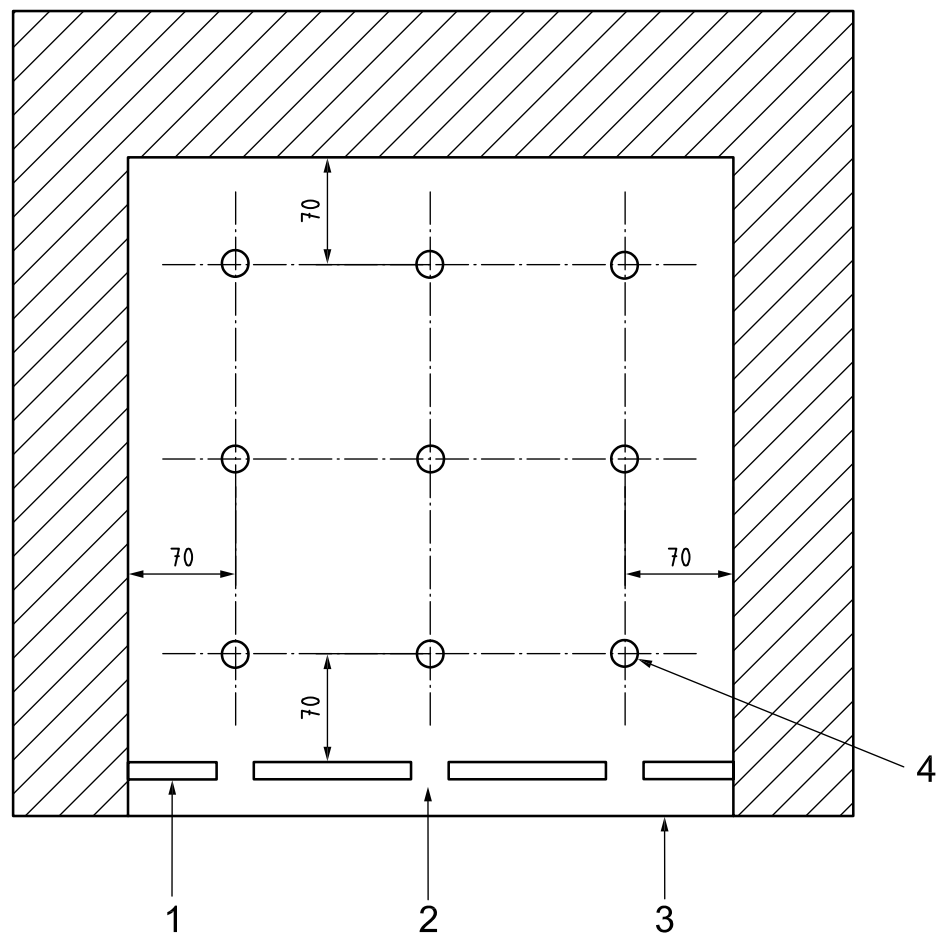
A.3.4 Operate the oven and, inserting the anemometer sensor through each aperture in turn, measure the air speed at all nine positions indicated in Figure A.1. Keep the gap between the plate and the stem of the anemometer airtight.

A.3.5 Read the maximum value of the air speed at each position so as to avoid any effect due to the directionality of the sensor.

A.4 Calculation of result

A.4.1 Calculate the mean value of the air speed measured at the nine measurement positions.

Dimensions in millimetres



Key

- 1 plastic plate
- 2 aperture
- 3 door opening
- 4 measurement position

Figure A.1 — Positions for measuring air speed in oven

Annex B (informative)

Precision

B.1 General

Two interlaboratory test programmes (ITPs) and the precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. The first ITP was organized in 1996 and the results analysed in 1997, and the second one in 2005. Consult ISO/TR 9272 for precision concepts and nomenclature. Annex C gives guidance on the use of repeatability and reproducibility results.

B.2 Precision details of the first ITP

B.2.1 Prepared test pieces were sent out to all participating laboratories using four compounds (of types NR, NBR, EPDM and AEM). Ageing was carried out by both method A and method B.

The ageing time was 168 h for all compounds, at 70 °C for NR, 100 °C for NBR, 125 °C for EPDM and 150 °C for AEM.

B.2.2 A total of 16 laboratories participated in this ITP. Eleven of the laboratories carried out the ageing by method A and ten laboratories by method B. Five of the laboratories used both method A and B. For certain of the tests carried out after ageing, values were missing from the compiled data, and for these tests fewer than these numbers of laboratories were involved. The actual number for each test is listed in the precision tables.

B.2.3 The hardness was measured in accordance with ISO 48:1994¹⁾, method M, before and after ageing. The three tensile strength properties were measured in accordance with ISO 37 on five test pieces before and after ageing. Type 1 and type 2 dumb-bell test pieces were used.

B.2.4 The precision determined in this ITP is a type 1 precision, i.e. fully prepared test pieces were submitted to all laboratories. The precision is also an intermediate-term or intermediate time period precision, with a time of two to three weeks between the two replicate determinations. This is in distinction to the more usual day 1 to day 2 replication with a few days between the determinations.

The symbols used in the tables are as follows:

r = repeatability, in measurement units;

(r) = repeatability, expressed as a percentage of the average;

R = reproducibility, in measurement units;

(R) = reproducibility, expressed as a percentage of the average.

(r) and (R) have only been calculated for all the materials together.

1) Withdrawn since the ITP was carried out (currently valid edition ISO 48:2010).

B.3 Precision results from the first ITP

B.3.1 The precision results are given in Tables B.1 to B.4 for method A (low air speed) and in Tables B.5 to B.8 for method B (high air speed). In these tables, no values of the relative precision (r) and (R) are given for the individual materials because many of the mean values of the performance parameters are near zero and this gives very large (r) and (R) values that have little meaning. The tables do give a mean value (similar but not equal to a pooled value) for all four materials together. These overall means are useful in comparing the relative precision of the four types of test performed. The relative precision for these overall means enables the two methods (A and B) to be compared.

B.3.2 On reviewing the tables, it will be observed that there is only a small difference between the repeatability r and the reproducibility R , and in several cases the two are equal. This phenomenon has been observed in previous ISO 188 ageing-precision testing. This demonstrates that a very large component of the variation observed in this type of testing is not due to differences between laboratories, but is due to some inherent source of variation that is just as likely to occur “within” a laboratory as on a “between”-laboratory basis. This unknown source is connected with the ageing process.

**Table B.1 — Ageing precision determined from change in hardness (IRHD)
(method A: low air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	3,1	3,10		3,63		11
NBR	4,4	2,08		3,68		11
EPDM	22,0	5,50		10,30		11
AEM	3,9	6,78		7,78		11
Absolute mean (without regard to sign)	8,3	4,4		6,3		
Relative precision			53		76	

**Table B.2 — Ageing precision determined from change in tensile strength (TS_b)
(method A: low air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	-8,7	8,43		9,34		11
NBR	6,6	9,26		11,83		11
EPDM	4,1	8,24		14,92		11
AEM	-9,3	8,13		10,71		11
Absolute mean (without regard to sign)	7,2	8,5		11,7		
Relative precision			118		162	

**Table B.3 — Ageing precision determined from change in stress at 100 % elongation (S_{100})
(method A: low air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	25,2	13,4	43	16,0	65	11
NBR	38,4	26,8		26,8		11
EPDM	247,1	78,9		135,3		11
AEM	0,4	15,4		22,7		11
Absolute mean (without regard to sign)	77,7	33,6		50,2		
Relative precision						

**Table B.4 — Ageing precision determined from change in elongation at break (E_b)
(method A: low air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	−13,3	10,36	38	10,36	50	11
NBR	−17,7	14,00		14,00		11
EPDM	−66,5	4,85		7,44		11
AEM	0,8	7,72		17,12		11
Absolute mean (without regard to sign)	24,2	9,2		12,2		
Relative precision						

**Table B.5 — Ageing precision determined from change in hardness (IRHD)
(method B: high air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	4,1	5,14	30	5,14	49	10
NBR	8,7	3,20		5,29		10
EPDM	35,9	3,89		9,67		10
AEM	8,0	5,04		8,00		10
Absolute mean (without regard to sign)	14,2	4,3		7,0		
Relative precision						

**Table B.6 — Ageing precision determined from change in tensile strength (TS_b)
(method B: high air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	-8,5	7,07	122	9,23	134	10
NBR	12,3	12,88		12,88		10
EPDM	7,9	11,88		11,88		10
AEM	-4,4	8,93		10,73		10
Absolute mean (without regard to sign)	8,3	10,2		11,2		
Relative precision						

**Table B.7 — Ageing precision determined from change in stress at 100 % elongation (S_{100})
(method B: high air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	24,3	10,3	22	14,0	51	10
NBR	54,4	25,0		26,7		10
EPDM	392,1	62,5		194,0		10
AEM	19,3	12,0		14,1		10
Absolute mean (without regard to sign)	122,5	27,4		62,2		
Relative precision						

**Table B.8 — Ageing precision determined from change in elongation at break (E_b)
(method B: high air speed)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR	-14,8	6,86	29	9,65	39	10
NBR	-19,3	9,41		13,14		10
EPDM	-73,0	5,76		8,89		10
AEM	-3,3	9,39		11,80		10
Absolute mean (without regard to sign)	27,6	7,9		10,9		
Relative precision						

B.4 Precision details of the second ITP

B.4.1 Prepared test pieces were sent out to all participating laboratories using three compounds (of types NR, NBR and EPDM). Ageing was carried out in type 1 and type 2 ovens using method B.

The ageing time was 72 h and 168 h for all compounds at 85 °C for NR, 100 °C for NBR and 125 °C for EPDM.

B.4.2 A total of 11 laboratories participated in this ITP. Five of the laboratories carried out the ageing in type 1 ovens and six laboratories in type 2 ovens. The actual number of laboratories for each test is listed in the precision tables.

B.4.3 The three tensile strength properties were measured in accordance with ISO 37 on five test pieces before and after ageing. Type 1A test pieces were used. Hardness was omitted from the analysis because there were insufficient test results.

B.4.4 The precision determined in this ITP is a type 1 precision, i.e. fully prepared test pieces were submitted to all laboratories. The precision is also an intermediate-term or intermediate time period precision, with a time of two to three weeks between the two replicate determinations. This is in distinction to the more usual day 1 to day 2 replication with a few days between the determinations.

The symbols used in Tables B.9 to B.14 are the same as those for the first ITP.

B.5 Precision results from the second ITP

B.5.1 The precision results are given in Tables B.9 to B.11 for type 1 ovens and in Tables B.12 to B.14 for type 2 ovens. In these tables, the values for the two ageing times, 72 h and 168 h, are included, but no values of the relative precision (r) and (R) are given for the individual materials, as in the first ITP. The relative precision for these overall means enables the two types of oven to be compared in the same way as in the first ITP.

B.5.2 On reviewing the tables, it can be seen that the type 1 and type 2 ovens give almost the same precision. The type 2 oven in fact gives slightly more uniform ageing and a slightly larger change in the properties on ageing.

**Table B.9 — Ageing precision determined from change in tensile strength (TS_b)
(type 1 oven)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR, 72 h	−3,2	4,2		8,7		5
NR, 168 h	−11,5	6,7		15,7		5
NBR, 72 h	0,5	6,0		13,8		5
NBR, 168 h	−4,0	11,6		11,3		5
EPDM, 72 h	−6,0	7,7		10,3		5
EPDM, 168 h	−7,8	14,9		19,0		5
Absolute mean (without regard to sign)	5,5	8,5	155	13,1	238	
Relative precision						

**Table B.10 — Ageing precision determined from change in stress at 100 % elongation (S_{100})
(type 1 oven)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR, 72 h	26,6	30,0		30,8		5
NR, 168 h	45,6	54,1		45,7		5
NBR, 72 h	39,5	7,4		48,5		5
NBR, 168 h	52,1	8,2		59,7		5
EPDM, 72 h	78,3	44,5		58,0		5
EPDM, 168 h	102,5	48,0		78,2		5
Absolute mean (without regard to sign)	57,4	32,0	56	53,5	93	
Relative precision						

**Table B.11 — Ageing precision determined from change in elongation at break (E_b)
(type 1 oven)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR, 72 h	-12,5	11,9		9,6		5
NR, 168 h	-19,3	1,4		13,8		5
NBR, 72 h	-23,0	4,7		15,6		5
NBR, 168 h	-29,3	9,1		13,1		5
EPDM, 72 h	-42,8	5,9		4,2		5
EPDM, 168 h	-49,3	13,3		11,4		5
Absolute mean (without regard to sign)	29,4	7,7	26	11,3	38	
Relative precision						

**Table B.12 — Ageing precision determined from change in tensile strength (TS_b)
(type 2 oven)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR, 72 h	-4,4	7,5		5,5		6
NR, 168 h	-16,1	9,4		9,5		6
NBR, 72 h	-6,7	7,8		17,2		6
NBR, 168 h	-9,6	7,3		8,2		6
EPDM, 72 h	-9,5	7,2		10,3		6
EPDM, 168 h	-9,6	12,7		14,0		6
Absolute mean (without regard to sign)	9,3	8,7	94	10,8	116	
Relative precision						

**Table B.13 — Ageing precision determined from change in stress at 100 % elongation (S_{100})
(type 2 oven)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR, 72 h	38,4	31,5		24,5		6
NR, 168 h	59,1	36,7		29,8		6
NBR, 72 h	53,7	10,4		24,7		6
NBR, 168 h	75,0	28,5		28,9		6
EPDM, 72 h	88,2	27,4		32,2		6
EPDM, 168 h	112,1	39,6		59,6		6
Absolute mean (without regard to sign)	71,1	29,0	41	33,3	47	
Relative precision						

**Table B.14 — Ageing precision determined from change in elongation at break (E_b)
(type 2 oven)**

Material	Mean change %	Within laboratory		Between laboratories		Number of labs
		r	(r)	R	(R)	
NR, 72 h	−15,6	13,8		10,2		6
NR, 168 h	−26,1	13,0		10,2		6
NBR, 72 h	−29,6	9,1		12,7		6
NBR, 168 h	−36,0	1,6		3,9		6
EPDM, 72 h	−47,9	14,5		14,7		6
EPDM, 168 h	−53,2	10,4		16,9		6
Absolute mean (without regard to sign)	34,7	10,4	30	11,4	33	
Relative precision						

Annex C (informative)

Guidance for using precision results

C.1 The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. without regard to sign).

C.2 Enter the appropriate precision table (for whatever test parameter is being considered) at an average value (of the measured parameter) nearest to the “test” data average under consideration. This line will give the applicable r , (r) , R or (R) for use in the decision process.

C.3 With these r and (r) values, the following general repeatability statements may be used to make decisions:

- a) For an absolute difference: the difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability r on average not more than once in twenty cases.
- b) For a percentage difference between two test (value) averages: the percentage difference

$$\frac{|x_1 - x_2|}{\frac{1}{2}(x_1 + x_2)} \times 100$$

between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability (r) on average not more than once in twenty cases.

C.4 With these R and (R) values, the following general reproducibility statements may be used to make decisions:

- a) For an absolute difference: the absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility R not more than once in twenty cases.
- b) For a percentage difference between two test (value) averages: the percentage difference

$$\frac{|x_1 - x_2|}{\frac{1}{2}(x_1 + x_2)} \times 100$$

between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility (R) not more than once in twenty cases.

Annex D **(normative)**

Calibration schedule

D.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration is carried out in the “as-received” condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

D.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table D.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a more specific or detailed calibration procedure than in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

- P particular procedure;
- C requirement to be confirmed, but no measurement;
- N initial verification only;
- S standard interval as given in ISO 18899;
- U in use.

Table D.1 — Calibration frequency schedule

Parameter	Requirement	Clause or subclause in ISO 18899:2004	Verification frequency guide	Notes
Oven size	Such that volume of test piece does not exceed 10 % of free space	C	U	
Provision for suspending test pieces	At least 10 mm apart and (for cabinet and forced air circulation ovens) at least 50 mm from sides	15.2	U	
Temperature	Within tolerances specified in Clause 8	18 (procedure B)	S	
Material	No copper or copper alloys used in construction of heating chamber	C	N	
Air flow	Between 3 and 10 changes per hour	16.3	S	
Cell-type oven				
Height	300 mm minimum	15.2	N	
Air speed	Shall depend only on exchange rate	C	N	
Design	Cells surrounded by good heat transfer medium and air passing through one cell shall not enter other cells	C	N	
Cabinet oven	No fans in heating chamber	C	N	
Forced air circulation oven				
Layout	See 4.1.4	C	N	
Air speed	Between 0,5 m/s and 1,5 m/s (type 1) or 0,5 m/s \pm 0,25 m/s (type 2)	P	S	See Annex A for a method of verification

In addition to the items listed in Table D.1, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

- a timer;
- a thermometer for monitoring the conditioning and test temperatures.

Bibliography

- [1] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*
- [2] ISO 11346, *Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use*

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